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DEMILITARIZATION PLANS: OPERATION OF THE CHEMICAL AGENT MUNITION--ETC(U)
MAR 77 S LAWHORNE

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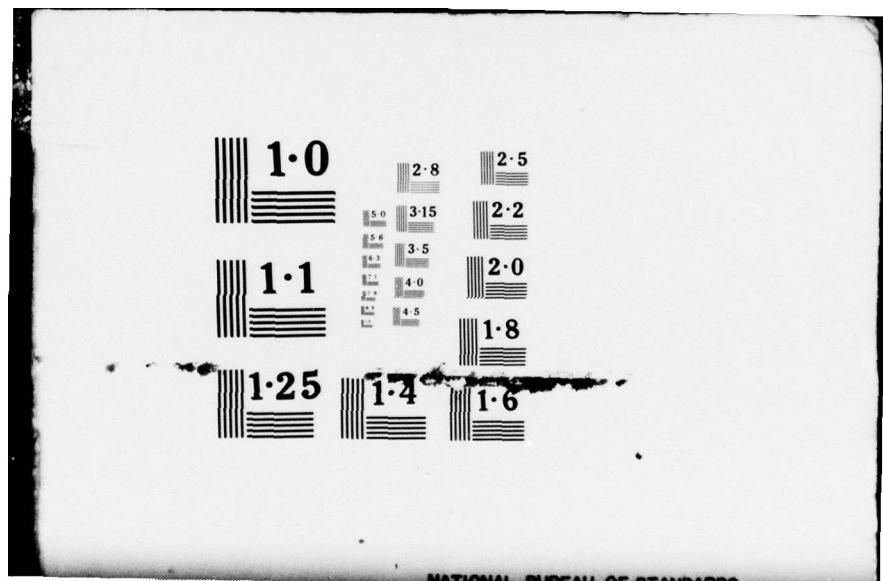
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(6) DEMILITARIZATION PLAN
OPERATION
OF THE
CHEMICAL AGENT MUNITIONS DISPOSAL SYSTEM
(CAMDS)

AT
TOOELE ARMY DEPOT, Inclosure Number 2,
Statistically Significant
Sampling Program.

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INCLOSURE NO. 2

STATISTICALLY SIGNIFICANT SAMPLING PROGRAM

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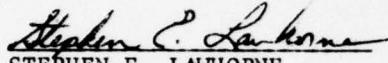
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Glue

Statistically Significant Sampling Program
Test Summary
ADS-ST-H, HT, GB, VX-TEAD (South)-1-1 thru 3

26 June 1974

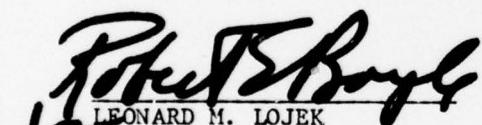
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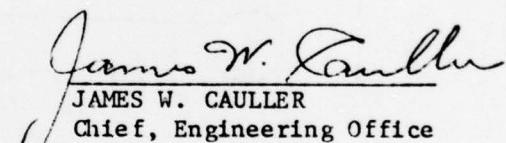

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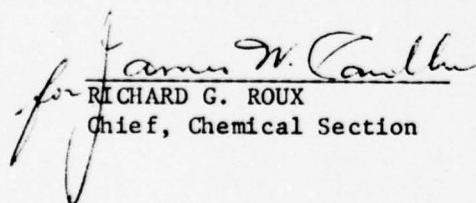
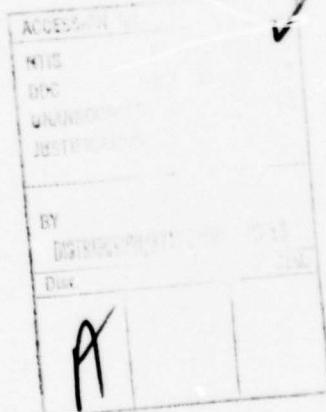

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I. INTRODUCTION

The present chemical compositions of the agents within stockpiled munitions and the internal pressure of these munitions was determined by sampling a statistically significant number of each type of munition.

A total of 321 munitions were sampled: 160 mustard (H, HT) munitions, 45 VX munitions, and 116 GB munitions. Testing and sampling included measurement of torque required to remove lifting plugs from mustard munitions, measurement of internal pressure within munitions, collection of agent samples, and evaluation of the agent's chemical and physical condition. Only 67 mustard munitions were pressure tested and agent sampled.

As an additional part of the program, forty 155mm mustard projectiles were drained as completely as possible of their agent fill, and the sludge filled projectiles were shipped to Rocky Mountain Arsenal for use in Metal Parts Furnace Pilot Tests.

The tests were performed in the South Area of Tooele Army Depot from April to July 1973 following the Statistically Significant Sampling Program Test Plan, March 73.

II. OBJECTIVES

- A. Determine the force required to remove the nose closure devices from stockpiled munitions.
- B. Determine the distribution of aluminum and bakelite fuze cups in stockpiled mustard projectiles.
- C. Determine whether or not bursters could be removed from projectiles with undue force.
- D. Determine the internal pressure in stockpiled munitions.
- E. Characterize the agent in stockpiled munitions by determining:
 1. agent purity
 2. impurities
 3. gross upper and lower limits of the impurities
- F. Drain forty 155mm mustard projectiles as completely as possible of their agent fill, and transfer the projectiles to RMA for use in the Metal Parts Furnace Pilot Tests.

III. NARRATIVE TEST DESCRIPTION AND DISCUSSION

A. Nose Closure and Burster Removal Tests.

The force required to remove the lifting plugs from projectiles presently in storage is information necessary to the design of various CAMDS components. The ease with which the explosive burster could be removed was also of interest to determine the extent of sticking bursters.

Seventy (70) M104 and M110 155mm projectiles were tested, and ninety (90) M2 4.2 in. projectiles were tested. The torque required to remove the lifting plugs from these projectiles was recorded in foot-pounds in the remarks column of Data Sheet 1.

Fuze cup material was recorded for the M104's and M110's. Bakelite fuze cups were originally installed in the projectiles, but they were replaced with aluminum fuze cups during regular maintenance operations.

Once the lifting plugs and fuze cups were removed from the M104's and M110's, the ease with which the bursters could be removed was tested. During regular maintenance operations, the burster wells were painted to prevent rust. The possible sticking of the burster due to the paint was anticipated. If the burster could not be removed readily by the force of gravity alone, the projectile was returned to storage for use in further testing at a future date.

The data from these tests were recorded on Data Sheet 1 and are presented in Inclosure 1.

Those projectiles selected for pressure and agent sampling were transferred to the work igloo, and all other projectiles were returned to storage.

B. Pressure and Agent Sampling.

The pressure and agent sampling were conducted in igloo 2212 in the South Area of Tooele Army Depot. The same general procedure was followed for each munition sampled, although exact details varied with the individual munition.

Each munition was handled within a glove box inside the igloo in accordance with TEAD SOP's. The glove box and the igloo were maintained at negative pressure with M6 filters, which insured containment of all agent vapors. The operations were performed by USA Technical Escort Center personnel.

All projectiles were clamped in place and sealed in a glove box. An air powered drill was used to drill a 7/16" hole in the side wall of the projectile. Any internal pressure within the projectile was directed about the drill bit, through part of the drill housing, and read by a pressure gauge before being vented into decon solution. After disengaging the drill fixture, the hole was tapped to accomodate a 1/4" NPT pipe plug. Two agent samples of approximately 40 mls. each were transferred from the projectile to glass culture tubes by means of a pipet. The culture tubes were sealed with Teflon lined screw-on caps and wrapped with Parafilm. The projectile was sealed with a Teflon coated 1/4" NPT pipe plug, cleaned, and placed in an approved and tested container. After stenciling complete identification information on the outside of the storage container, the projectile was placed in isolated storage as a Code H munition.

A slight departure from this procedure in the case of mustard filled 155mm M104's and M110's was the addition of a thawing step. These projectiles were heated with infra red lamps to a skin temperature of 100°F before proceeding with the sampling.

M55 rockets required a different procedure because they are stored in their fiberglass shipping container, and in the case of Code H rockets, a steel identification kit. The rocket, shipping container, and steel identification kit were placed in the glove box before it was sealed. The rocket was removed only far enough from the shipping container to clamp it in place under the drill. The rocket was drilled; the pressure was read and vented; and the agent was sampled with a pipet. The hole was tapped and plugged with a Teflon coated 1/4" NPT pipe plug. The pipe plug was tightened flush with the skin of the warhead and then covered with Aerobond 2244 epoxy, manufactured by Northwest Plastics, Inc. of Seattle, Washington. The rocket was then replaced in its original shipping container, which was placed in a steel I.D. kit, stenciled, and placed in isolated storage as a Code H munition. This procedure left the rockets in a form suitable to demil in CAMDS rather than creating a need for a special demilitarization operation.

M23 land mines also required a modified sampling procedure. The mines are stored in cans of three (3) each. The mines were removed from the storage can and sealed in the glove box one at a time. The initiator well was removed, and the agent samples were transferred by pipet to the glass culture tubes. The initiator well was replaced to seal the mine, and the mine was cleaned, removed from the glove box, and replaced in its original can. The mines were then placed in isolated storage as Code H munitions.

Data Sheets 4 (see Inclosure II) were used to record all data collected during this phase of the program. As the munitions were

logged for sampling, they were assigned to an identification number consisting of a letter prefix for the agent type and a number denoting its order in the sampling program, i.e. H1, H2 . . . , V1, V2 . . . This identification number was used to identify the munition through the ensuing sampling procedures and to identify the agent samples collected from the munition.

The internal pressure recorded on Data Sheet 4 is the pressure read from the gauge in the glove box. This pressure has been corrected to reflect the actual pressure found within the munition rather than that read after pressurizing the drill assembly. The correction procedure and a table of the adjusted pressures are presented in Inclosure III.

C. Mustard Draining Operations.

The 155mm M104's and M110's were drained as completely as possible of their liquid mustard fill, leaving only sludge in the projectiles, so they could be used in CAMDS Metal Parts Furnace pilot tests.

Several approaches to the draining were tried before identifying the most successful method. Methods attempted included use of a vacuum line inserted in the original sampling hole and applying a vacuum to the entire projectile.

In the most successful approach the projectiles were heated to 100°F to thaw the mustard before fixing them in place and sealing them in a glove box. A second 7/16" hole was drilled and tapped 180° from the original sampling hole, the projectile was rotated 180°, and the pipe plug was removed from the original hole. The mustard drained by gravity into a funnel shaped pan from which it was transferred by vacuum through 1/2" I.D. tubing into a ton container. When the gravity flow ceased, 1 psi positive pressure was applied to the top hole in the projectile to insure the liquid flow had stopped rather than being blocked. The positive pressure often forced significant amounts of mustard from the projectile after the gravity flow had ceased. After draining, the holes were sealed with pipe plugs and the projectile decontaminated. The projectiles, which had been weighed prior to draining, were weighed again after draining. The weight difference was taken as the amount of mustard drained from the projectile.

The amount of mustard remaining in the projectiles after draining was determined in representative projectiles by inserting a known quantity of solvent, thoroughly agitating the projectile, and withdrawing the solvent. Chemical analysis then showed the amount of mustard remaining in the projectile. On the basis of the chemical analyses, a mean of 37.59 mls. of mustard remained per projectile.

Data Sheets 5 were used to record all data collected during this phase of the program, and they are presented in Inclosure IV. Note that the same identification numbers are used on the projectiles as assigned during the sampling phase.

D. Agent Shipments.

The agent samples collected in the sampling operation at TEAD were transferred to Dugway Proving Ground (DPG) either for chemical analysis in the laboratories or as an intermediate point in their transfer to Edgewood Arsenal for use in detoxification studies.

Two (2) shipments of mustard agent in solvent were sent to DPG for analysis to verify complete draining of the 155mm mustard projectiles.

All agent shipments were escorted by personnel from the USA Technical Escort Center.

The dates, quantities, and end points of each agent shipment are included in Inclosure V.

E. Chemical Analysis of Agent Samples.

The Chemical Laboratories at Dugway Proving Ground performed analyses of the chemical agent samples to characterize the agent as stored in stockpiled munitions. The parameters and methods of analysis are listed for each agent:

1. Mustard (H & HT)

- a. density by pycnometer or specific gravity balance (every other sample).
- b. viscosity by calibrated Ostwald viscometers (every other sample).
- c. heat of combustion by adiabatic calorimetry.
- d. metals screening by emission spectroscopy.
- e. quantitative analysis for major metals by atomic absorption spectroscopy (to include total iron).
- f. purity by MIL-M-12051A.
- g. total chlorine by argentimetric titration.

h. total sulfur by titration with lead salt.

i. total carbon and hydrogen by automatic C, H & N analyzer.

j. soluble and insoluble iron by water extraction.

k. heat capacity by adiabatic calorimetry (5 samples).

2. VX

a. density by pycnometer or specific gravity balance (every other sample).

b. viscosity by calibrated Ostwald viscometers (every other sample).

c. heat of combustion of adiabatic calorimetry.

d. metals screening by emission spectroscopy.

e. quantitative analysis for major metals by atomic absorption spectroscopy (to include total iron and aluminum).

f. purity by MIL-C-51105 (MV) (to include free mercaptan and bis-VX).

g. CV by wet analysis.

h. total phosphorous by molybdenum blue procedure.

i. quantitative analysis of stabilizer by gas chromatography.

j. total sulfur by titration with lead salts.

k. total carbon, hydrogen, and nitrogen by automatic C, H & N analyzer.

l. heat capacity by adiabatic calorimetry (5 samples).

3. GB.

a. density by pycnometer or specific gravity balance (every other sample).

b. viscosity by calibrated Ostwald viscometers (every other sample).

c. heat of combustion by adiabatic calorimetry.

- d. metals screening by emission spectroscopy.
- e. quantitative analysis for major metals by atomic absorption spectroscopy (to include total iron and aluminum).
- f. purity by DPG procedure.
- g. total phosphorous by molybdenum blue procedure.
- h. free fluoride by specific ion electrode.
- i. quantitative identification of stabilizer by gas chromatography.
- j. total carbon and hydrogen by automatic C, H & N analyzer.
- k. DIMP and TBA by gas chromatography.
- l. heat capacity by adiabatic calorimetry (5 samples).

A copy of the Summary Report from the Dugway Proving Ground Chemical Laboratories giving their analytical results is included as Inclosure VI.

IV. EVALUATION

A. Nose Closure and Burster Removal.

A maximum torque of 150 foot-pounds was required to remove the lifting plug from a sample lot of ninety (90) 4.2" projectiles. That torque was 3 standard deviations from the mean of 76.833 foot-pounds, indicating a 99% probability that the torque requirement for each projectile in the entire group will be less than or equal to 150 foot-pounds.

For a sample lot of seventy (70) 155mm mustard projectiles (M104's and M110's) a maximum torque of 40 foot-pounds was required to remove the lifting plug. This torque was 3.8 standard deviations from the mean of 11.429 foot-pounds, indicating a greater than 99% probability that the torque requirements for each projectile in the entire group will be less than or equal to 40 foot-pounds.

Examination of the seventy (70) M104's and M110's revealed that 15.7% of the sampled projectiles still had bakelite fuze cups. Equipment must be designed to remove both the bakelite and aluminum fuze cups.

Three (3) bursters were found in a sample lot of seventy (70) (4.3%) that could not be removed by gravity. No further investigation was made during this program to determine the extent to which these bursters were stuck. The three (3) projectiles were marked and set aside for future testing.

B. Pressure and Agent Sampling.

Pressure sampling showed internal pressures in only the H filled M104's and M110's and GB filled M55 rockets.

The internal pressures in the mustard projectiles were greater than those in the rockets, with the highest sampled pressure being 88.3 PSIG (adjusted). The mean for the sample lot of 47 projectiles was 20.59 PSIG with a standard deviation of \pm 21.57. The probability is slightly greater than 99% (3.1 standard deviations) that the internal pressure in each mustard projectile will be less than or equal to 88.3 PSIG. Note that these projectiles were heated to a skin temperature of 100°F to thaw the mustard.

For a sample lot of 23 M55 GB rockets, the highest internal pressure was 46.3 PSIG (adjusted), which is 4.01 standard deviations

from the mean of 4.096 PSIG. Using this sample size, there is a greater than 99% probability that the internal pressure in each rocket will be less than or equal to 46.3 PSIG.

The agent sampling revealed the condition of the agent in the stockpiled munitions. With the exception of H type mustard, most of the agent (HT, VX, GB) was free flowing. There was a small amount of suspended solid matter in the collected samples. The HT samples were pale yellow, and the VX samples were clear to yellow. The GB samples were amber to tea colored to yellow in color.

Two (2) GB rockets sampled from lot 1033-55-1076 showed a definite variation from the normal. The agent from these rockets was almost a jelly - very similar to some of the mustard samples in consistency. Internal pressures of 19.8 and 46.3 PSIG accompanied the unusual agent. The lot history revealed no reason for the variation in agent characteristics. The rockets, manufactured in April 1965, were filled with agent from lot 255, which was just stabilized with TBA and later restabilized with DICDI. These two (2) agent samples were scheduled for special analyses to explain the variance.

Several code H M55 GB rockets were leaking from holes in the warhead as large as 1/16" in diameter. In these cases, a considerable amount of agent was running free in the fiberglass shipping containers leaving a reddish film on the outside of the aluminum warhead.

H-type mustard filled projectiles presented sampling problems due to the consistency of the agent. Even after attempting to thaw the mustard agent, samples were hard to collect through pipets due to the high solids content of the agent. In one instance no more than 15 mls. of sample could be collected from a full projectile. The mustard varied in color from dark brown to black.

C. Mustard Draining.

The mustard filled M104's and M110's were drained for use in Metal Parts Furnace pilot tests at RMA. The draining operation revealed a great deal of useful information about the mustard agent and proposed CAMDS procedures.

One procedure being considered at that time involved draining mustard from the projectiles by a probe under vacuum. Attempts to simulate such a procedure ended in a complete failure due to the high solids content, even after heating the projectiles to 100°F.

In all alternate approaches the solids presented the greatest difficulties. Increasing pipe size reduced the problem somewhat, but bends, joints, and valves still served as collecting points for the solid material and quickly clogged the lines. While only a 1/3 horsepower vacuum pump with a capacity of 5 cfm was used to induce flow in the mustard lines, the extent of the difficulties promised trouble even under better pumping conditions.

The data collected during the draining operation shows very vividly the amount of solid material contained in the projectiles. An average of 2.9 lbs was removed from the projectiles (including the 80 ml sample) which were originally filled with 11.7 pounds of mustard. The total amount of mustard drained varied from 0.73 lbs for one projectile to 10.36 lbs for another, indicating that the total solids remaining in the projectiles ranged as high as 10.97 lbs.

E. Chemical Analysis of Agent Samples.

Chemical analysis of the agent samples was used to define the average condition of the agent in the stockpiled munitions as well as the range encountered in the impurities detected.

Suspended solid matter was present in the liquid agent samples. The solid portion of the sample was removed by centrifuge or filtration, and only the liquid portion was analyzed. In type H mustard, the solids represented a significant portion of the sample. The solids in the other agents were relatively insignificant.

The heat of combustion analysis performed on mustard samples (H, HT) is indicated as yielding approximate results since the combustion products etched the interior of the calorimeter bomb adding to the heat to be measured. Error limit was estimated to be \pm 2%.

In mustard samples the purity was determined by freezing point depression. For HT mustard, this analysis gives the molar purity of HD mustard. HT mustard was mixed as 70% HD and 30% T by weight.

The agent samples were screened for the presence of metals on a Fisher Duo-Spectranal. The metals detected in this test were then analyzed quantitatively by atomic absorption spectroscopy. The soluble iron, however, was analyzed by wet chemistry.

VX samples were analyzed qualitatively to confirm the presence of DICDI stabilizer, then quantitatively to determine the stabilizer concentration.

GB samples were also analyzed qualitatively to identify the stabilizer before quantitative analysis to determine the concentration. Presence of only TBA indicates preroundout or roundout agent. Presence of only DICDI indicates redistilled agent. Presence of both stabilizers (DICDI and trace TBA) indicates restabilized agent.

The summary report submitted by DPG is included as Inclosure VI. Attached to this report are the calculated mean values for each parameter. Note sample G105 was disregarded because decon solution was spilled into the sample during packaging.

In the mustard samples (H, HT) the sulfur concentration is higher than the theoretical value while the chlorine concentration is less than the theoretical value. The chlorine may have been lost as a gaseous product of decomposition, and would have contributed to the internal pressures experienced after heating the H mustard projectiles. The sulfur concentration would appear higher due to the loss of the chlorine in the liquid sample. The manufacturing process may have left excess sulfur in the solution, also. The iron content in HT mustard (81.79 ppm mean) indicates some attach by the agent upon the steel projectile, because low iron HD was used in the original manufacture of the agent. The much higher iron concentration in H mustard (3527.08 ppm mean) is due in large to the manufacturing process which did not involve distillation. The majority of the iron (2987.50 ppm mean) is soluble. Heat of combustion and density of the mustard samples did not vary greatly from the theoretical values. Note that these analyses apply only to the liquid portion of the agent samples. Analysis of the mustard sludge will be conducted in conjunction with the Metal Parts Furnace pilot tests.

Analysis of VX samples detected virtually no metals. Even samples from M55 rockets showed no aluminum present. A mean of 1.188 ppm of iron was detected in the samples with a maximum of 4.3 ppm. Density and heat of combustion of the samples parallel theoretical values, but the viscosity is about 2 centipoises higher than the theoretical value.

Metals played a more prominent role in the GB samples. The mean iron content was 89.68 ppm, while the M55 rocket samples showed a mean of 45.59 ppm iron. Erosion of the steel munitions is indicated by this difference. The aluminum content in the samples averaged 1686.5 ppm, compared to the average of 1765.2 ppm for the M55 rockets. Erosion of the aluminum warhead by the agent is supported by this difference in aluminum contents. The increasing number of leaking rockets being discovered in storage facilities also supports the

conclusion that GB agent attacks the aluminum warheads. The jelly-like agent discovered in the two rockets sampled from lot 1033-55-1076 is not satisfactorily explained by the results of the analyses performed thus far. Additional work must be done to further characterize these two samples and explain their variation from the characteristics exhibited by the other samples. Note that the viscosity reported for sample G97 was calculated using the filtered portion of the sample. Other parameters tested paralleled the theoretical values expected.

Table 1 presents a comparison of the theoretical values available to the test results obtained.

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Summary of Analyses vs Theoretical Values

TABLE 1

<u>Parameter</u>	<u>Theoretical Value</u>	<u>Sample Mean</u>	<u>Standard Deviation (+)</u>	<u>High Sample</u>	<u>Low Sample</u>
A. Agent HT	Purity (as % HD)	40.94	4.62	49.3	32.5
	Freezing Point (°C)	-7.29	1.70	-4.21	-10.39
	Total Carbon (%)	33.51	1.13	38.2	33.0
	Total Hydrogen (%)	5.62	0.27	6.8	5.7
	Total Sulfur (%)	22.36	4.77	36.9	17.9
	Total Chlorine (%)	35.32	2.82	35.6	25.3
	Aluminum (ppm)	<0.1	0	<0.1	<0.1
	Total Iron (ppm)	0	81.79	22.20	128
	Soluble Iron (ppm)	0	45.63	11.51	67
	Insoluble Iron (ppm)	0	36.26	17.55	83
B. Agent H	Heat of Combustion (cal/g)	5036	4925.11	29.24	4970
	Density (g/ml @ 25°C)	1.26	1.2536	5.528 × 10 ⁻³	1.2587
	Viscosity (centipoise @ 25°C)	-	6.808	8.066 × 10 ⁻²	6.91
	Purity (as % HD)	100	67.69	5.17	79.0
	Freezing Point (°C)	14.0	2.44	1.86	6.72
B. Agent H	Total Carbon (%)	30.199	27.41	1.18	28.8
	Total Hydrogen (%)	5.069	4.75	0.17	5.2
	Total Sulfur (%)	20.156	26.40	1.45	29.1
	Total Chlorine (%)	44.57	37.43	2.45	42.2
	Total Iron (ppm)	0	3527.08	387.43	5100
	Soluble Iron (ppm)	0	2987.50	385.72	3500
	Insoluble Iron (ppm)	0	539.58	375.72	2100
	Heat of Combustion (cal/g)	4676	4395.45	37.51	4500
	Density (g/ml @ 25°C)	1.2685	1.301	4.042 × 10 ⁻³	4300
	Viscosity (centipoise @ 25°C)	-	5.026	0.2874	5.67

TABLE 1 cont'd

<u>Parameter</u>	<u>Theoretical Value</u>	<u>Sample Mean</u>	<u>Standard Deviation (\pm)</u>	<u>Low Sample</u>	<u>High Sample</u>
				<u>Low Sample</u>	<u>High Sample</u>
C. Agent GB					
Purity (%)	100	82.23	6.85	96.9	60.8
Total Phosphorus (%)	22.109	18.166	1.58	21.4	14.0
Total Carbon (%)	34.293	36.84	1.07	39.1	32.4
Total Hydrogen (%)	7.195	7.535	0.285	8.3	6.6
Total Nitrogen (%)	-	0.414	0.263	1.7	0.0
Free Fluoride (%)	-	0.5693	0.4639	3.07	0.10
TBA (%)	-	-	-	2.5	<0.1
DICDI (%)	-	-	-	0.9	<0.1
DIMP (%)	-	1.536	0.8401	4.6	<0.1
Total Iron (ppm)	0	89.68	51.32	200	3
Aluminum (ppm)	0	1686.5	475.35	2600	220
Heat of Combustion (cal/g)	-	5278.91	108.307	5508	5069
Density (g/ml @ 25°C)	1.0887	1.0791	8.174×10^{-3}	1.873	1.0577
Viscosity (centipoise @ 25°C)	1.28	1.64	0.163	2.00	1.30
D. Agent VX					
Purity (%)	100	79.53	4.59	86.0	68.4
Total Phosphorus (%)	12.23	9.32	.6572	10.8	8.2
Total Sulfur (%)	12.66	12.31	2.90	22.1	8.1
Total Carbon (%)	47.41	49.01	7.50	57.7	42.3
Total Hydrogen (%)	9.55	9.141	0.550	10.7	8.0
Total Nitrogen (%)	5.53	5.44	0.4035	6.1	4.3
CV (%)	-	4.61	1.93	11.7	2.3
DICDI (%)	-	0.2195	0.2589	0.73	<0.02
Free Mercaptan (%)	-	0.4716	0.3985	2.86	0.17
Bis-VX (%)	-	2.068	1.489	9.77	0.20
Total Iron (ppm)	0	1.188	0.680	4.3	0.7
Aluminum (ppm)	0	<1.0	0	<1.0	<1.0
Heat of Combustion (cal/g)	7402	7421.96	27.01	7490	7377
Density (g/ml @ 25°C)	1.008	1.0062	4.824×10^{-3}	1.0138	0.9967
Viscosity (centipoise @ 25°C)	9.96	11.8565	1.0212	13.52	10.48

STATISTICALLY SIGNIFICANT SAMPLING PROGRAM

INCLOSURES I-VI

Inclosures I through VI containing raw data sheets, sample calculations, and schedules have been removed from this printing.

Copies of these Inclosures are available on request to the Project Manager for Chemical Demilitarization and Installation Restoration, DRCPM-DRD-TM, Aberdeen Proving Ground, MD 21010